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## EXECUTIVE SUMMARY

# CARBON-14 CHARACTERIZATION - INTERIM REPORT

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Chemical Research Department

Although it was predicted that substantial quantities of carbon-14 would be produced in a nitrogen gas annulus system by the $^{14}N(n,p)^{14}C$ reaction, the formation of a solid, easily dispersible form of this long-lived beta-emitting isotope was not anticipated. Hence, early in the Large Scale Fuel Channel Replacement (LSFCR) project, a carbon-14-bearing particulate material was inadvertently released in the vaults of Pickering Units 1 and 2. The Chemical Research Department was mandated by the Carbon-14 Task Group to characterize this particulate form of carbon-14 to provide data for an evaluation of the associated decontamination, health and safety, and waste disposal issues. This report contains a summary of the results of studies on numerous samples of the material collected from different locations and during different phases of the LSFCR project. The following physical and chemical properties were investigated; particle size, morphology, specific activity, chemical composition, solubility, volatility, and chemical speciation. A summary of the typical characteristics of the material in each type of sample is shown in the attached table.

The studies to date have revealed that the carbon-14-bearing material has appeared as airborne particulate in the size range of 1 to 2 microns (mean aerodynamic diameter), a gaseous form which is mainly $^{14}CO_2$, and as surface contamination on tools that have been directly in contact with the 'native' material in the reactor or on surfaces contaminated by airborne particulate. Depending on the degree of 'contamination' of the carbon-14-bearing material with other materials, it generally has high specific activity ranging from 0.1 Ci/g to 1.0 Ci/g, and it consistently contains carbon-12 plus carbon-14, and nitrogen, and when iron is present as an intimate mixture, iron oxide. A small fraction of the carbon-14-bearing material is soluble (passes through a

0.1 micron filter) in saline and dilute acids, while the majority of the material ( >80%) is essentially insoluble and inert to common chemical reagents. Similarly, a small (2-10%) volatile carbon-14-bearing component is associated with the solid material which can be volatilized under vacuum at 300°C. Freshly exposed material emanates activity mainly as $^{14}CO_2$ and $^{14}CO$.

There has not been any positive identification of chemical species in the carbon-14-bearing material; however, it is possible to rule out some compounds such as pure elemental carbon or an iron-carbon compound. The high specific activity (1 Ci/g) 'top hat' sample is being studied in great detail. The results of Auger, X-ray photoelectron and pyrolysis mass spectrometry are currently being evaluated and suggest that the active top hat material is a $^{14}CN(H)$ polymer which undergoes partial surface oxidation and liberates $^{14}CO_2$ and $^{14}CO$ slowly at room temperature and rapidly above 200°C.

Future work should be directed towards confirmation of the suspected speciation of the carbon-14 in the Pickering gas annulus deposits by spectroscopic techniques or classical functional group analysis, and to further elucidate the degradation mechanism and determine the ramifications for decontamination and waste disposal.

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Summary of Characteristics of Carbon-14-containing Material

| Sample Type | Airborne Particulate from Shock Heating | | End Fitting Component Swipes | | Annulus Gas Bellows Debris | Calandria Swipe | "Top Hat" | Annulus Gas Pigtail Filters |
|---|---|---|---|---|---|---|---|---|
| | Unit 1 | Unit 2 | Unit 1 | Unit 2 | Unit 1 | Unit 1 | Unit 2 | Unit 1 |
| C-14 Activity in Total sample | 1 uCi | 1 uCi | 1 mCi | 1 mCi | 10 uCi | 50 uCi | 10 mCi | 2 uCi |
| Cobalt-60 Activity in Total Sample | 50 nCi | 1 nCi | 30 nCi | 30 nCi | 2 nCi | 3 uCi | 3 uCi | 30 nCi |
| C-14 Specific Activity Ci/g of sample | 0.04 | 0.04 | 0.02 | 0.07 | 0.04 | 0.04 | 1.0 | 0.001 |
| Chemical Composition | | | | | | | | |
| Carbon (wt %) | 17 | 45 | 15 | 30 | 20 | 40 | 40 | 27 |
| Nitrogen (wt %) | 3 | 9 | 2 | 2 | 2 | - | 20 | - |
| Oxygen (wt %) | 19 | 15 | 25 | 15 | 22 | 15 | 12 | 20 |
| Iron (wt %) | 42 | 25 | 50 | 53 | 56 | 35 | 28 | 40 |
| C-14/C-12 (Approx %) | 5 | 2 | 3 | 5 | 5 | 3 | >50 | 0.1 |
| Solubility (% of C-14) | | | | | | | | |
| in Saline | 3 - 19 | 20 | - | - | 23 | - | 23 | 10 |
| in 0.1M HCl | 13 | - | - | - | 5 | - | 10 | 10 |
| Volatility in vacuo up to 300°C (% C-14) | 2 | - | - | - | - | - | 10 | - |
| Particle Size, Morphology, Additional Information | 1 - 5 um; heavily contaminated with iron oxide | ~2 um (Andersen plate); on filters, ≤10 um with "Xmas tree" shapes | ~9 um, heavily contaminated with iron oxide | | heavily contaminated with cadmium oxide and iron oxide | heavily contaminated with iron oxide and zirconium oxide | >10 um "Xmas trees" and other dendritic shapes | heavily contaminated with iron oxide |

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To: Dr. D. Mills
Director of Research

## Carbon-14 Characterization - Interim Report

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The Chemical Research Department was mandated by the Carbon-14 Task Group to characterize the particulate form of carbon-14 found in the Pickering Unit 1 & 2 annulus gas system to provide data for an evaluation of the associated decontamination, health and safety, and waste disposal issues. It has been found that, depending on the degree of 'contamination' of the carbon-14-bearing material with other materials, it generally has high specific activity ranging from 0.1 Ci/g to 1.0 Ci/g, and it consistently contains carbon-12 plus carbon-14, and nitrogen, and when iron is present as an intimate mixture, iron oxide. A small fraction of the carbon-14-bearing material is soluble (passes through 0.1 micron filter) in saline and dilute acids, while the majority of the material ( >80%) is essentially insoluble and inert to common chemical reagents. Similarly, a small (2-10%) volatile carbon-14-bearing component is associated with the solid material which can be volatilized under vacuum at 300° C. Freshly exposed material emanates activity mainly as $^{14}CO_2$ and $^{14}CO$. Current results support the hypothesis that the material is a $^{14}CN(H)$ polymer which undergoes partial surface oxidation and liberates $^{14}CO_2$ and $^{14}CO$ slowly at room temperature and rapidly above 200°C.

### INTRODUCTION

In March 1985, the Analytical Services Section of the Chemical Research Department received samples of material which emitted low energy radiation, from the Pickering NGS Units 1 & 2 retubing project. Initially, the request was to determine the nature of the material and the source of it's radioactivity. Preliminary tests on these initial samples showed they contained about 400-600 microcuries of carbon-14 per gram, the carbon-14 component was essentially inert (insoluble and non-volatile) and 50% of the material was carbon. Subsequent samples were submitted in order to develop accurate, reliable methods of detecting and quantifying the amount of carbon-14 on various samples from personnel protective devices (Personnel Air Sampler (PAS) filters) or from area monitoring (air sampler filters and surface swipes). The method adopted was combustion of the sample with collection and

liquid scintillation counting of the combustion products, and is reported in Research Division Report C85-44K /1/.

With the formation of the Carbon-14 Task Group, the role of the Chemical Research Department was further defined to encompass the characterization of the physical and chemical properties of the carbon-14-bearing material. This was to include determination of the size and specific activity, physical morphology, chemical and radionuclide composition, volatility and solubility, and, to whatever extent possible, to determine the chemical species present in the material and it's method of formation /2/.

Preparations at the Research Division Radioactive Materials Laboratory included identification of designated areas and modification of glove box(es), as well as development of handling procedures and radiation protection procedures for working with the highly mobile, high specific activity samples. The procedures and radiation monitoring results are described in references 3, 4, & 5.

Most of the characterization work was conducted at the Ontario Hydro Research Laboratory although a limited amount of work was performed at AECL's Chalk River Nuclear Laboratory /6,7,8/, and at the University of Western Ontario's Surface Science Laboratory /9/.

Obtaining a clear description of the physical and chemical nature of the carbon-14-bearing material has been very difficult due to a number of factors. First and foremost, the high specific activity coupled with the low energy of the beta emission from carbon-14 has made working with the material difficult in the extreme - necessitating minuscule sample sizes and work frequently had to be performed at the detection limits of analytical techniques. Secondly, a large number of samples have been examined over the period of the study, gathered from diverse locations and during different retubing operations, which generally exhibited different characteristics, particularly between certain Unit 1 and Unit 2 samples. Finally, the breadth of the field of carbon chemistry and the universally poor understanding of most of the solid carbon systems like soot or rare polymers has meant that identification of the carbon species present in these samples is essentially original research.

The following sections contain general descriptions of the properties of the material, based on the samples tested to date, and the variation of these properties between samples. Detailed descriptions of the samples, their origin, physical and chemical properties, the analytical techniques employed and the confidence in the conclusions are contained in the references. Readers are urged to consult the references for exact information about the samples. It should also be noted that the understanding of the material's properties has increased as the investigation progressed, and the analytical techniques have been refined over time, so the information in earlier references is less comprehensive than that in later references. The final section contains a summary of the current understanding of the nature of the carbon-14-bearing material and the future planned work to further characterize the material.

## RESULTS

### Samples

The different origin of the samples - which unit, the location in the unit, the retubing operation underway when the sample was obtained, the method of sample

collection and sample substrate, all have lead to observed differences in the gross sample characteristics and in the individual carbon-14-bearing particle characteristics, due to 'contamination' of the material with other material in the vault and because in most sample collection operations the sample was disturbed and hence not truly representative of what was originally present in the reactor. While in some cases the 'contaminated' sample was truly representative of the material that was causing problems during operations, such as the airborne material released to the vault during shock heating, the extraneous material was an interference since it lead to confusion about which particles were carbon-14-bearing material, what the actual chemical composition of the carbon-14-bearing material was, and what the true specific activity of the carbon-14-bearing particles was, since it was apparent that the 'contaminated' samples were heterogeneous mixtures of particles from different locations.

The samples are grouped broadly by their origin in the following, together with references which describe in detail the sample description and the analytical results obtained.

End-Fitting Removal Samples - Unit 1, March/85
Original samples included brushes used as a precaution to remove any misplaced garter springs. Subsequent samples included swipes from the end-fitting removal trough extension and a swipe that had been suspended in liquid scintillation cocktail /10,11/.

Airborne Particulate Sampled during Shock Heating - Unit 2, April 27/85
Particulate sample collected by filtering vault air through a millipore filter during a shock heating operation.

Airborne Particulate and Gas Sampled during Shock Heating - Unit 1, May 25-30/85
Particulate and gaseous sampling of the air in the vault in the vicinity of the open fuel channel and on the rolling shield during shock heating. Four such runs were performed for channels H8, H17, G7, & H9. These samples consisted of airborne particulate filters, Andersen sampler plates, sodium hydroxide filled bubblers and carbon cartridges collected during shock heating activities in Unit 1 /11,12,13/.

End-Fitting Body Samples - Unit 2, September 1985
Swipes were taken from the outer surface of the K11E pressure tube and end-fitting bodies which had been stored under-water at Chalk River Nuclear Laboratories since removal in 1983 /11/.

Annulus Bellows Debris - Unit 1, October 1985
These samples were taken by vacuuming loose material from several annulus bellows on the west reactor face of Unit 1 /11/.

Calandria Swipes - Unit 1, January 1986
These samples consisted of a pair of sponges which had been pushed through channel A13 of Unit 1 as part of a post-pressure tube removal cleaning operation /11/.

Pigtail Debris - Unit 1, December 1985 - January 1986
Samples of loose debris were collected in pigtail flow verification filters from tests on the east and west reactor face of Unit 1 /14/.

Calandria Tube Protective Sleeve ("Top Hat") Unit 2 - April 1986

A heavily contaminated calandria tube protective sleeve, generally referred to as a "top hat," used in pressure tube removal operations in Unit 2, was sent to research for examination in April 1986 /14/.

**Characterization Results**

A summary of the characteristics of the different samples is given in Table 1. More details about the sample characteristics and the references are given in the following paragraphs.

**Particle size**

Information on the particle size of the carbon-14-bearing particles is relevant to such issues as health and safety, waste disposal and environmental monitoring; these in turn require data on the filterability of the carbon-14-bearing particulate suspended in water or air. An Andersen impactor was used to study the size of particles suspended in vault air by the shock heating process. Optical and electron microscopy confirmed the results reported by J. Stephenson /13/ showing a mean aerodynamic diameter in the range of 1 or 2 microns.

Microfiltration of carbon-14-bearing particulate taken from an end-fitting removal trough extension and suspended in water gave a mean particle diameter of about 9 microns /10/.

**Particle Morphology**

A considerable amount of work has been carried out on the morphology of the carbon-14-bearing particles using optical and electron microscopy /10,11,14/. Although many particles have been photographed from a wide variety of samples, work has been hampered by uncertainties in what are active or inactive particles. For this reason autoradiography has been used to reveal the activity of microparticles. The most active, and morphologically interesting, particles have been found in Unit 2 samples from shock heating or on the calandria tube protective sleeve (top hat). These particles show a characteristic branched (dendritic) structure and have been dubbed as "Christmas trees" /10,14/. It appears that these particles were at one time attached to surfaces in, or close to, the reactor core and have been broken off by retubing operations.

**Specific Activity**

A determination of the carbon-14 specific activity in a sample is a measure of the purity of the carbon-14. A specific activity of 4.5 Ci/g implies 100 % pure carbon-14. Samples received prior to April 1986 exhibited carbon-14 specific activities less than 0.1 Ci/g and were typically in the range of 0.05 Ci/g (based on total sample weight). However, measurements on material from a Unit 2 top hat have resulted in carbon-14 specific activities approaching 1 Ci/g of sample or 20% pure carbon-14. Most samples exhibit a slight gamma activity mainly from Co-60; however, its specific activity is generally less than 1/1000th of the C-14 specific activity /11,14/.

**Solubility**

At the request of Safety Services Department, the solubility of carbon-14-bearing species in simulated body fluids was studied. The solubility in saline solution varied from nearly 3% (for samples of airborne dust obtained during the shock heating of Channel H8 Unit 1) to 23 % for most other samples. Similar results were obtained by leaching with 0.1 M hydrochloric acid /15,16/.

Long-term leach tests showed that there is an initial rapid dissolution of the carbon-14-bearing species followed by a very slow increase in the leachate activity /16/.

The solubility of the carbon-14-bearing species in a sample of deposit on an end-fitting body from Channel K11 of Unit 2 (which had been stored under water for almost 2 years) was studied. The active material was found not to be soluble in strong acids, concentrated ammonium hydroxide and in many organic solvents. A considerable amount of labware was contaminated during this experiment and an attempt was made to decontaminate it. A mixture of surfactants was found to be effective /17/.

The solubility of carbon-14-bearing species in the vault air absorbed on carbon cartridges during shock heating operations was studied in different solvents in order to obtain some insight into the nature of the material. The active species were soluble in polar solvents like water, alcohol and acetone but much less soluble in non-polar solvents like hexane, toluene or diethyl ether. The polar nature of the gaseous samples was therefore established /17/.

**Volatility**

A carbon-14-bearing species emanates from samples on standing in air, and the emanating species can be trapped in alkaline solutions. At room temperature very little activity emanates (at a maximum **initial** rate of 0.01%/h). The emanation rate increases with temperature but **decreases with time** /14,15/. It appears that radiolytically enhanced oxidation of the sample to $^{14}CO$ and $^{14}CO_2$ occurs at room temperature. Thermal oxidation to $^{14}CO_2$ (mainly) occurs rapidly in air at elevated temperatures with a maximum **initial** rate of approximately 4%/h at 200°C and approximately 8%/h at 300°C. The reaction mechanism and kinetics are complex and an analysis of the data is in progress to determine if identification of the reaction kinetics can provide further clues to the chemical speciation of the material.

A part of the activity can be volatilized under vacuum at 300°C and can be trapped on a cold trap. This volatile fraction contains between 2-10% of the initial carbon-14 activity /15,17/.

**Chemical Composition**

A determination of the elemental composition of the samples shows what elements are "diluting" the carbon-14 and simultaneously provides clues to the environment in which the solid carbon-14 containing material was formed. The elements carbon, nitrogen, oxygen and iron have consistently been found in all the samples; excluding the top hat sample, their compositions are in the range of 15-45% carbon, 2-9% nitrogen, 15-25% oxygen and 25-56% iron /11/. None of the samples contain particles composed only of elemental carbon. Only in the

top hat samples have iron-free particles been routinely identified; these particles appear to contain mainly carbon and nitrogen although data for hydrogen or deuterium have not been obtained /14/.

The saline and acidic leachates were analyzed for chemical composition by distillation and three fractions were defined. The inorganic fraction (consisting of carbonates, bicarbonates and low-boiling organics which could be distilled out of an acid medium) accounted for between 5-70 % of the soluble carbon-14 activity; the organic fraction (consisting of material oxidizable by aqueous persulphate ion to carbon dioxide) contained 20-60 % of the activity and the balance was an unoxidizable fraction which was resistant to the oxidizing agent during the period of the experiment /16/.

Air sampling was carried out in the Unit 1 vault during shock heating. Approximately 98% of the gaseous carbon-14 was trapped in a NaOH bubbler and was presumed to be $^{14}CO_2$. The remaining 2% gaseous carbon-14 was adsorbed on an activated carbon cartridge and consisted of 30% inorganic carbon-14 with the rest being organic carbon-14. The polar nature of the adsorbed material was demonstrated by its solubility properties /17/.

**Chemical Speciation**

All of the samples received by the Research Division were microscopic and on a matrix which made direct speciation impossible. Isolation or preconcentration techniques were therefore employed to examine this aspect. The volatile fraction which could be trapped on a cold finger was examined using Fourier transform infrared spectroscopy. Only carbon-hydrogen bonds could be identified. The absorption signal was extremely weak and no further information could be obtained nor could it be determined whether the signal was due to impurities in the sample /17/.

Speciation of the aqueous extract of the activity absorbed on the carbon cartridge was attempted using ion chromatography. However, excessive impurities on the commercial activated carbon used in the cartridges saturated the chromatographic column and useful information could not be obtained /17/.

There are many hypotheses about the chemical form of the carbon-14 and the compound in which it is contained. While a large number of analytical techniques have been applied to the study, most of the results have yielded little information which can support any one compound. However, it is possible to rule out some compounds such as pure elemental carbon or an iron-carbon compound. The chemical and physical properties of the material, such as its inertness, thermal stability (to 300$^{o}$C) in vacuum, while being combustible in air or oxygen, could indicate a polymeric material (i.e. $-^{14}CN(H)-$), and many carbon/nitrogen polymers exhibit properties akin to the carbon-14-bearing material. Researchers studying carbon-14 "hot-atom" chemistry frequently produce polymeric materials that they are unable to speciate and they generally refer to them simply as polymeric materials. The results of Auger, X-ray photoelectron and pyrolysis mass spectroscopic investigations are currently being evaluated. The analysis of mass spectral data, which is usually capable of providing unambiguous information on chemical speciation, is complicated by the equality of the C-14 and N-14 masses. However, the results to date still support the hypothesis that the active material is a $^{14}CN(H)$ polymer and occurs in its purest form in the top hat sample. It also appears that the active material undergoes partial surface oxidation (perhaps enhanced by self-

radiolysis), forming $>^{14}C=O$ groups which are liberated as $^{14}CO_2$ and $^{14}CO$ slowly at room temperature and rapidly above 200°C.

**Current Status**

A summary of the typical characteristics of the material in each type of sample is shown in the attached table. Note that these are representative results from a number of analyses of subsets of the sample and that the appropriate reference should be consulted for more details.

The studies to date have revealed that the carbon-14-bearing material has appeared as airborne particulate in the size range of 1 to 2 microns (mean aerodynamic diameter), a gaseous form which is mainly $^{14}CO_2$, and as surface contamination on tools that have been directly in contact with the 'native' material in the reactor or on surfaces contaminated by airborne particulate. Depending on the degree of 'contamination' of the carbon-14-bearing material with other materials, it generally has high specific activity ranging from 0.1 Ci/g to 1.0 Ci/g, and it consistently contains carbon-12 plus carbon-14, and nitrogen, and when iron is present as an intimate mixture, iron oxide. A small fraction of the carbon-14-bearing material is soluble in saline and dilute acids (the solubility being defined as the material which passes through 0.1 micron filter), while the majority of the material ( >80%) is essentially insoluble and inert to common chemical reagents. Similarly, a small (2-10%) volatile carbon-14-bearing component is associated with the solid material which can be volatilized under vacuum at 300° C. Freshly exposed material emanates activity mainly as $^{14}CO_2$ and $^{14}CO$.

The speciation work so far has not resulted in any positive results partly due to the small, low specific activity samples under investigation and due to contaminants associated with the samples. The top hat sample shows a high specific activity (1 Ci/g) and therefore is being studied in great detail. The results of Auger, X-ray photoelectron and pyrolysis mass spectrometry are currently being evaluated and suggest that the active top hat material is a $^{14}CN(H)$ polymer which undergones partial surface oxidation and liberates $^{14}CO_2$ and $^{14}CO$ slowly at room temperature and rapidly above 200°C.

Future work should be directed towards confirmation of the suspected speciation of the carbon-14 in the Pickering gas annulus deposits by spectroscopic techniques or classical functional group analysis, and to further elucidate the

degradation mechanism and determine the ramifications for decontamination and waste disposal.

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Table 1

Summary of Characteristics of Carbon-14-containing Material

| Sample Type | Airborne Particulate from Shock Heating | | End Fitting Component Swipes | | Annulus Gas Bellows Debris | Calandria Swipe | "Top Hat" | Annulus Gas Pigtail Filters |
|---|---|---|---|---|---|---|---|---|
| | Unit 1 | Unit 2 | Unit 1 | Unit 2 | Unit 1 | Unit 1 | Unit 2 | Unit 1 |
| C-14 Activity in Total sample | 1 uCi | 1 uCi | 1 mCi | 1 mCi | 10 uCi | 50 uCi | 10 mCi | 2 uCi |
| Cobalt-60 Activity in Total Sample | 50 nCi | 1 nCi | 30 nCi | 30 nCi | 2 nCi | 3 uCi | 3 uCi | 30 nCi |
| C-14 Specific Activity Ci/g of sample | 0.04 | 0.04 | 0.02 | 0.07 | 0.04 | 0.04 | 1.0 | 0.001 |
| Chemical Composition | | | | | | | | |
| Carbon (wt %) | 17 | 45 | 15 | 30 | 20 | 40 | 40 | 27 |
| Nitrogen (wt %) | 3 | 9 | 2 | 2 | 2 | - | 20 | - |
| Oxygen (wt %) | 19 | 15 | 25 | 15 | 22 | 15 | 12 | 20 |
| Iron (wt %) | 42 | 25 | 50 | 53 | 56 | 35 | 28 | 40 |
| C-14/C-12 (Approx %) | 5 | 2 | 3 | 5 | 5 | 3 | >50 | 0.1 |
| Solubility (% of C-14) | | | | | | | | |
| in Saline | 3 - 19 | 20 | - | - | 23 | - | 23 | 10 |
| in 0.1M HCl | 13 | - | - | - | 5 | - | 10 | 10 |
| Volatility in vacuo up to 300°C (% C-14) | 2 | - | - | - | - | - | 10 | - |
| Particle Size, Morphology, Additional Information | 1 - 5 um; heavily contaminated with iron oxide | ~2 um (Andersen plate); on filters, ≈10 um with "Xmas tree" shapes | ~9 um, heavily contaminated with iron oxide | | heavily contaminated with cadmium oxide and iron oxide | heavily contaminated with iron oxide and zirconium oxide | >10 um "Xmas trees" and other dendritic shapes | heavily contaminated with iron oxide |

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